

NASA TECHNICAL NOTE



NASA TN D-5640

c. 1

NASA TN D-5640

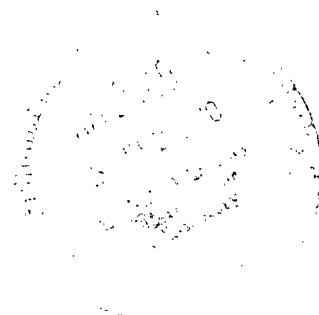


LOAN COPY: RETURN TO
AFWL (WLOL)
KIRTLAND AFB, N MEX

HIGH-STRENGTH TANTALUM COMPOSITE BY THERMOMECHANICAL WORKING

by Ruluff D. McIntyre

*Lewis Research Center
Cleveland, Ohio*



NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • JANUARY 1970



0132540

1. Report No. NASA TN D-5640	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle HIGH-STRENGTH TANTALUM COMPOSITE BY THERMOMECHANICAL WORKING	5. Report Date January 1970	6. Performing Organization Code
7. Author(s) Ruluff D. McIntyre	8. Performing Organization Report No. E-5201	10. Work Unit No. 129-03
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135	11. Contract or Grant No.	13. Type of Report and Period Covered Technical Note
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546	14. Sponsoring Agency Code	
15. Supplementary Notes		
16. Abstract The effect of thermomechanical processing by rolling on the tensile strength and micro-structural properties of a tantalum composite was studied. A core made of tantalum sheet and fibers embedded in a matrix of tantalum powder was rolled to very high levels of reduction that resulted in greatly improved strength at 77° F (298 K). The 30-vol. %-core composite had a strength of 322 000 psi (22.2×10^8 N/m ²), and the core had an apparent strength (extrapolated data) of 650 000 psi (44.82×10^8 N/m ²). The high strength appeared to be associated with a fine stable substructure generated by the thermomechanical working.		
17. Key Words (Suggested by Author(s)) Composite Refractory metal Thermomechanical working Tantalum	18. Distribution Statement Unclassified - unlimited	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 18
		22. Price* \$3.00

* For sale by the Clearinghouse for Federal Scientific and Technical Information
Springfield, Virginia 22151

HIGH-STRENGTH TANTALUM COMPOSITE BY THERMOMECHANICAL WORKING

by Ruluff D. McIntyre

Lewis Research Center

SUMMARY

An investigation was conducted to study the effect of thermomechanical processing on the tensile strength and microstructural properties of a tantalum composite. Tantalum sheet and fibers embedded in a matrix of tantalum powder were rolled to very high levels of reduction that resulted in greatly improved strength at 77° F (298 K). The strength of a 30-volume-percent composite at 77° F (298 K) was 322 000 psi (22.2×10^8 N/m²) after a 99-percent reduction in thickness. The matrix tantalum had a strength of 173 000 psi (11.93×10^8 N/m²). The 650 000-psi (44.82×10^8 N/m²) strength, obtained by extrapolation, for the sheet and fiber core was associated with a fine stable substructure in the core generated by thermomechanical working. The average cell, or barrier, dimension was well under a micrometer, which is small enough to cause considerable strengthening. The strength advantage of the composite over the matrix was retained to 1000° F (811 K). No strength difference existed between the composite and matrix at 1500° and 2200° F (1089 and 1478 K).

INTRODUCTION

Highly worked metals are usually much stronger than bulk metals. For example, drawn fibers may have strengths of over a half million psi (34.5×10^8 N/m²). The high strength of these materials can be related to a fine stable substructure that is a network of low-angle boundaries within the main crystals of a metallographic structure. Low-angle boundaries can serve as obstacles, or barriers, to dislocation movement. Such substructures are sometimes described as "fibrous" in appearance. A fiber-reinforced composite offers one way to start out with an artificially created fibrous structure having low-angle boundaries within the fibers. Enhancement of the fibrous structures can be achieved by using a thermomechanical process that involves work-anneal cycles. The method of extensive mechanical working of a fiber composite system to increase strength might possibly be applicable to many metal systems. Subjecting already highly

worked fibers and sheet to additional very high levels of reductions could be expected to result in further substantial gains in composite strength. The strength gains might be achieved without changing the chemical and heat-transfer properties of the component materials.

In the current investigation, tantalum materials were chosen because tantalum can be extensively cold worked at low temperatures. In addition, tantalum possesses unusually good heat-transfer and chemical properties for use in practical applications where higher strength would be an advantage. The high strength of the highly worked material would not be expected to be retained at high temperatures unless the barriers to dislocation movement remained stable. To make the composites, tantalum fibers and foil were embedded in powders and pressed into composites that were sintered at 4150°F (2561 K) in vacuum (5×10^{-5} torr or $6.65\times 10^{-3}\text{ N/m}^2$). The sintered composites were reduced approximately 80 percent in thickness at 2100°F (1422 K) and were rolled at 77°F (298 K) for final reduction in thicknesses (including the 80-percent reduction at 2100°F or 1422 K) of over 99 percent. Tensile strength, elongation, and percent of reduction in area of composites containing 5 to 30 volume percent of the highly worked tantalum reinforcements were obtained at temperatures ranging from 77° to 2200°F (298 to 1922 K). Optical and electron-microscopy techniques were used to assess microstructural properties.

MATERIALS AND PROCEDURE

Materials

Tantalum fibers, tantalum foil, and tantalum powder were the materials used to make the composites. The characteristics of these materials are described in table I.

Specimen Fabrication

Sheet bars weighing 225 grams and measuring 5.25 by 3.40 by 0.93 centimeter were pressed in a single-action die at a pressure of 30 ksi ($2.07\times 10^8\text{ N/m}^2$). Two types of specimens were prepared. One type was made from tantalum powder. The other type was made by aligning the fibers parallel to one another on tantalum foil and carefully rolling the fibers in the foil. The fibers wrapped in foil were then placed in the die and surrounded by the tantalum powder for pressing (see fig. 1). Some of the important processing conditions related to sintering and rolling of specimens are shown in table II along with density measurements. Sintering was done at 2000°F (1366 K) for 2 hours followed

TABLE I. - CHEMICAL ANALYSIS OF STARTING POWDER,
UNCONSOLIDATED CORE, AND FABRICATED COMPOSITE

Material	Condition	Chemical analysis	
		Element	Concentration, wt. %
Tantalum (matrix)	Powder, 4-micrometer diameter (by a subsieve sizer)	Oxygen	0.1010
		Carbon	.0040
		Nitrogen	.0114
		Hydrogen	.0028
		Tungsten	.0053
		Niobium	.0184
		Iron	.0050
		Molybdenum	.0030
		Tantalum	Balance
Tantalum (core)	Wire, 0.005-inch (1.27×10^{-4} -m) diameter; foil, 0.002 inch (0.51×10^{-4} m) thick	Oxygen	0.0050
		Carbon	.0025
		Nitrogen	.0020
		Hydrogen	.0030
		Tungsten	.0070
		Niobium	.0060
		Iron	.0010
		Tantalum	Balance
Composite	As sintered	Oxygen	0.1100
		Carbon	.0021
		Nitrogen	.0115
		Hydrogen	.0004
		Tungsten	.0069
		Niobium	.0195
		Copper	.0070
		Molybdenum	.0030
		Tantalum	Balance
Composite	After rolling at 2100 ⁰ F (1422 K)	Oxygen	0.1020
		Carbon	.0023
		Nitrogen	.0190
		Hydrogen	.0005
		Niobium	.0100
		Iron	.0020
		Tantalum	Balance
Composite	After final rolling at 77 ⁰ F (298 K)	Oxygen	0.0940
		Carbon	.0013
		Nitrogen	.0157
		Hydrogen	.0005
		Niobium	.0100
		Iron	.0010
		Tantalum	Balance

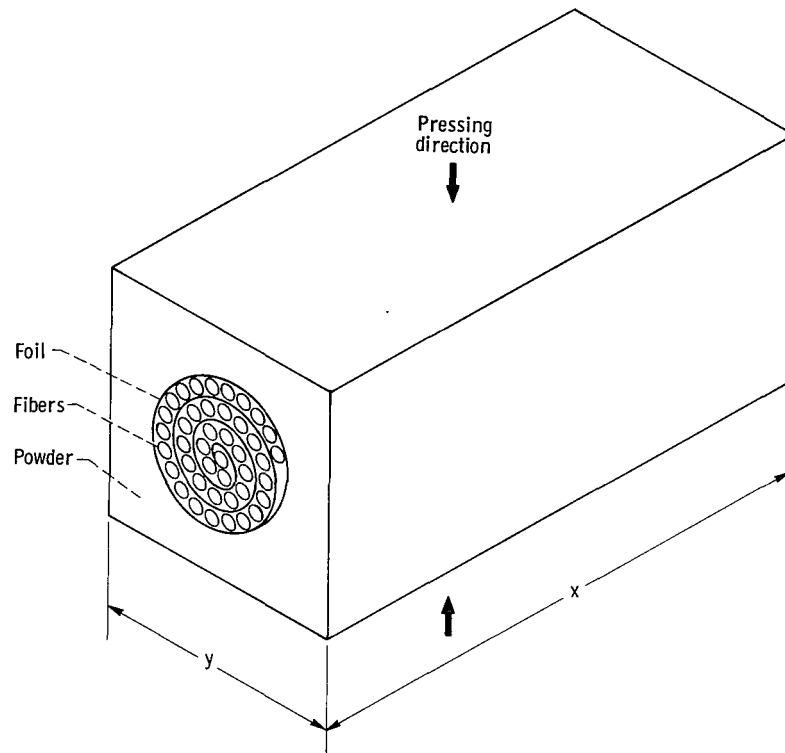


Figure 1. - Tantalum - tantalum-fiber composite before rolling.

TABLE II. - FABRICATION OF COMPOSITES

Condition	Temperature		Time, min	Atmosphere	Fiber thickness		Density, g/cm ³
	°F	K			in.	m	
Sintered ^a	2000	1366	120	Vacuum, 5×10^{-5} torr (6.65×10^{-3} N/m ²)	0.005	1.27×10^{-4}	13.6
	4150	2561	330				
Rolled ^b	2100	1422	75	Argon	0.001	0.254×10^{-4}	16.0
Rolled ^c	77	298	15	Air	0.00004	1×10^{-6}	16.4

^aAll composites were pressed in stainless-steel single-action hydraulic die at 30 ksi (2.07×10^8 N/m²) before sintering.

^b10-Percent reduction in thickness per pass for 80-percent total reduction at 2100° F (1422 K).

^c10-Percent reduction in thickness per pass to 0.001-in. (0.254×10^{-4} -m) final thickness.

by $5\frac{1}{2}$ hours at 4150° F (2561 K) in a vacuum of 5×10^{-5} torr ($6.65\times 10^{-3}\text{ N/m}^2$). After sintering, all specimens were wrapped in 0.002-inch- ($5.08\times 10^{-5}\text{-m-}$) thick tantalum foil and clad in 0.032-inch- ($8.13\times 10^{-4}\text{-m-}$) thick stainless-steel sheet. Seams were welded in argon to make closed cans around the specimens. The cans prevented the tantalum from oxidizing during elevated-temperature rolling.

All specimens were flat rolled first at 2100° F (1422 K) by using a 10-percent reduction in thickness per pass. The initial thickness of the compacts was approximately 0.380 inch ($9.65\times 10^{-3}\text{ m}$), and the final thickness was about 0.060 inch ($15.24\times 10^{-4}\text{ m}$). Clad specimens were heated to 2100° F (1422 K) in a furnace filled with argon before they were rolled in air. The stainless-steel cladding was removed from the specimens before rolling at 77° F (298 K) (room temperature). Room temperature rolling was done by using a 10-percent reduction in thickness per pass. The starting thickness was 0.060 inch ($15.24\times 10^{-4}\text{ m}$), and the final thickness was 0.001 inch ($2.54\times 10^{-5}\text{ m}$).

As a check on possible contamination, a chemical analysis was conducted after sintering, after rolling at 2100° F (1422 K), and after room temperature rolling. The values obtained appear in table I.

Several rolled sheet samples were heated in vacuum (5.0×10^{-5} torr) (or $6.65\times 10^{-3}\text{ N/m}^2$) at 1000° F (811 K) for 4 hours, at 1500° F (1089 K) for 1/2 hour, or at 2200° F (1478 K) for 3 hours to check the effect of heat treatment on strength.

Mechanical Property Testing

Tensile properties were investigated by using a tensile testing machine with a cross-head speed of 0.1 inch per minute (0.00254 m/min). Elongation results were obtained from crosshead motion. Test specimens had a 1-inch (0.0254-m) gage length with a reduced section that had a cross-sectional area which varied from 0.015 to 0.0005 square inch (9.677×10^{-6} to $0.323\times 10^{-6}\text{ m}^2$) depending on the amount of rolling prior to testing. The volume percent of reinforcement included in the cross section of a tensile bar was varied by machining away the matrix surrounding the fiber and foil core. Reduction-in-area values were obtained by measuring the cross-sectional area of the specimens before and after tensile testing. Elevated-temperature tensile tests were conducted in an evacuated chamber (5.0×10^{-5} torr or $6.65\times 10^{-3}\text{ N/m}^2$) equipped with a tantalum sleeve heater (ref. 1). Temperatures were measured with a platinum - platinum-13-percent-rhodium thermocouple and are estimated accurate to $\pm 20^{\circ}\text{ F}$ (11 K).

Metallographic Studies

Microstructures were examined at various magnifications to study fiber and foil size and distribution in the composites after rolling. An etchant composed of 50 parts sulfuric acid, 20 parts nitric acid, and 20 parts hydrofluoric acid was used for tantalum. The core size and structure in composites rolled to 0.001 inch (2.54×10^{-5} m) were studied by obtaining an electron micrograph at a magnification of 9500. A cellulose acetate replication technique was used.

A photograph was taken by electron transmission at a magnification of 70 000 to show the cellular substructure generated by the thermomechanical working process.

RESULTS

Mechanical Properties

Tensile properties. - Strength-related data for the materials studied appear in figures 2 to 4 and in table III. Included are ultimate tensile strength, reduction-in-area, and elongation data for specimens rolled to 80-, 95-, and 99-percent reductions in thickness. Tested were composites with 5-, 15-, and 30-volume percent reinforcement, as well as the unreinforced matrix. Testing temperatures ranged from 77° to 2200° F (298 to 1478 K).

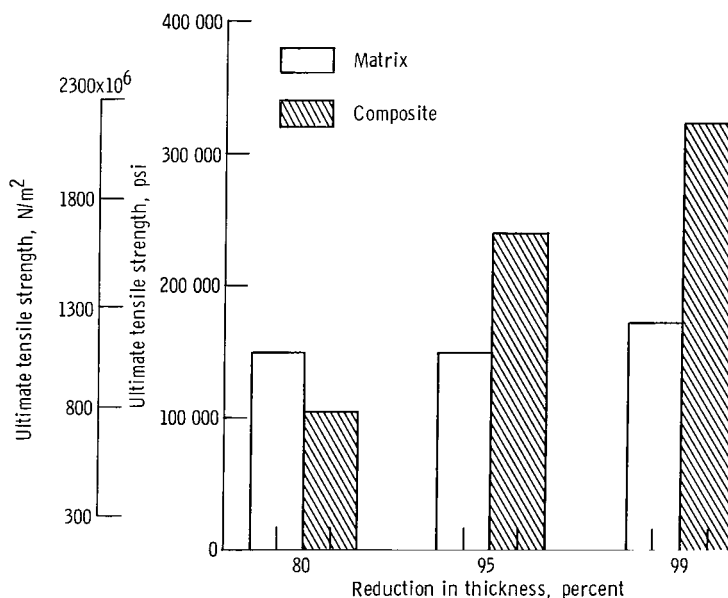


Figure 2. - Effect of rolling on ultimate tensile strength at 77° F (298 K) for matrix and 30-volume-percent composite.

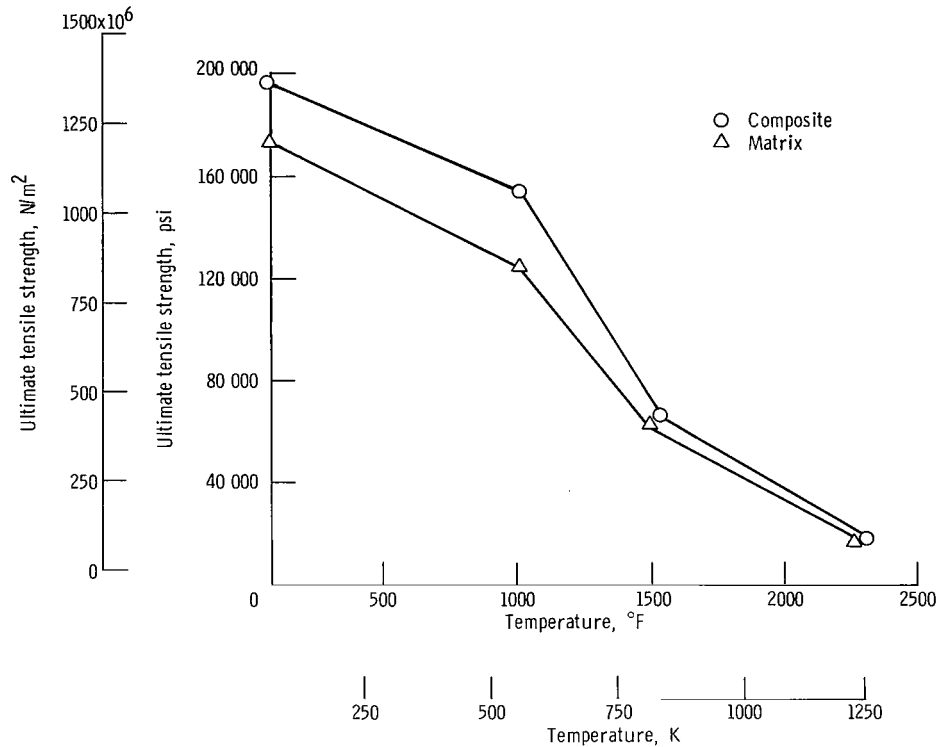


Figure 3. - Ultimate tensile strength of matrix and 15-volume-percent composite as function of temperature after 95-percent reduction in thickness by rolling.

Effects of rolling. - A comparison of strength values at 77° F (298 K) for the matrix and a composite with 30-volume percent reinforcement following 80-, 95-, and 99-percent reductions in thickness is presented in figure 2. The composite was weaker than the matrix after an 80-percent reduction. The composite had a strength of 102 000 psi (7.03×10^8 N/m²), whereas the matrix had a strength of 150 000 psi (10.34×10^8 N/m²). After a 95-percent reduction in thickness, the matrix retained its strength of 150 000 psi (10.34×10^8 N/m²). The composite strength increased to 240 000 psi (16.55×10^8 N/m²), which made it 90 000 psi (6.21×10^8 N/m²) stronger than the matrix. After a 99-percent reduction in thickness, the composite had a strength advantage of 149 000 psi (10.27×10^8 N/m²). The strength of the matrix was 173 000 psi (11.93×10^8 N/m²), whereas that of the composite was 322 000 psi (22.20×10^8 N/m²). Table III indicates that the matrix had a 23-percent reduction in area during tensile testing following an 80-percent reduction in thickness by rolling. The reduction in area for the matrix was 12 percent after a 95-percent reduction in thickness and was 1 percent after a 99-percent reduction in thickness. For a 30-volume-percent reinforced composite, the reduction in area was 36 percent after an 80-percent reduction in thickness by rolling. After 95- and

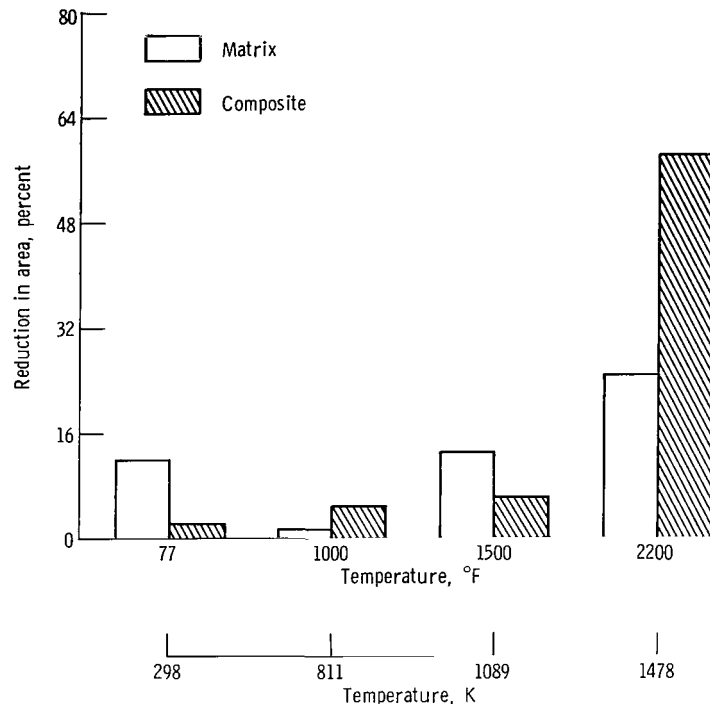


Figure 4. - Reduction in area of matrix and 15-volume-percent composite as function of test temperature after 95-percent reduction in thickness by rolling.

99-percent reductions in thickness, the composite showed 1 percent reduction in area during tensile testing at 77° F (298 K).

The variation of strength with temperature for a composite with 15-volume-percent reinforcement and for the matrix is shown in figure 3. After a 95-percent reduction in thickness, the composite is stronger than the matrix at 77° and 1000° F (298 and 811 K). The composite has the same strength as the matrix at 1500° and 2200° F (1089 and 1478 K).

Reduction-in-area properties as related to temperature. - Reduction in area is plotted against test temperatures in figure 4. A composite with 15-volume-percent reinforcement had low reduction-in-area values up to 1500° F (1089 K). A very pronounced increase in ductility was observed at 2200° F (1478 K). The reduction in area increased from 6 percent at 1500° F (1089 K) to a value of 58 percent at 2200° F (1478 K). The reduction in area for the matrix was greater at 77° and 1500° F (298 and 1089 K) relative to the composite. At 1000° and 2200° F (811 and 1478 K), the composite exhibited higher reduction-in-area values than the matrix. Figure 4 and table III show that the matrix had the lowest reduction-in-area value of all the materials tested and that this value occurred at a testing temperature of 1000° F (811 K). After an 80-percent reduction in thickness, the matrix had a reduction in area of 3 percent at 1000° F (811 K). After a 95-percent reduction in thickness, the reduction in area was 1 percent for the matrix.

TABLE III. - TENSILE DATA FOR INITIAL FOIL AND FIBERS, MATRIX, AND COMPOSITES

Composition	Condition	Test temperature		Ultimate tensile strength		Elongation, ^a percent	Reduction in area, percent	
		°F	K					
				psi	N/m ²			
Properties of initial foil and fibers								
Tantalum fibers, unalloyed	As drawn	77	298	119 000	8.2×10 ⁸	2	1	
Tantalum foil, unalloyed	Rolled to 99-percent reduction in thickness	77	298	116 000	8.0×10 ⁸	1	1	
Properties of matrix								
Tantalum matrix from powder	Rolled to 80-percent reduction in thickness	77	298	150 000	10.34×10 ⁸	5	23	
	Heat treated ^b	77	298	137 000	9.44	7	5	
	Rolled to 80-percent reduction in thickness	500	533	140 000	9.65	10	15	
	Rolled to 80-percent reduction in thickness	800	700	133 000	9.17	11	7	
	Rolled to 80-percent reduction in thickness	1000	811	114 000	7.86	8	3	
	Heat treated ^b	1000	811	108 000	7.45	10	7	
	Rolled to 80-percent reduction in thickness	1200	922	80 000	5.52	9	5	
	Rolled to 80-percent reduction in thickness	1500	1089	52 000	3.59	13	26	
	Heat treated ^b	1500	1089	37 000	2.55	14	16	
	Rolled to 80-percent reduction in thickness	2200	1478	17 000	1.17	25	65	
	↓	Rolled to 95-percent reduction in thickness	77	298	150 000	10.34×10 ⁸	1	12
			1000	811	127 000	8.76	13	1
			1500	1089	66 000	4.55	9	13
			2200	1478	180 000	1.24	12	25
	Rolled to 95-percent reduction in thickness	77	298	173 000	11.93×10 ⁸	1	1	
	Heat treated ^c	77	298	148 000	10.2×10 ⁸	1	1	
	Heat treated ^d	77	298	127 000	8.76×10 ⁸	1	1	

^aFrom crosshead motion.^bAt 2200° F (1478 K) for 3 hr.^cAt 1000° F (811 K) for 4 hr.^dAt 1500° F (1089 K) for 1/2 hr.

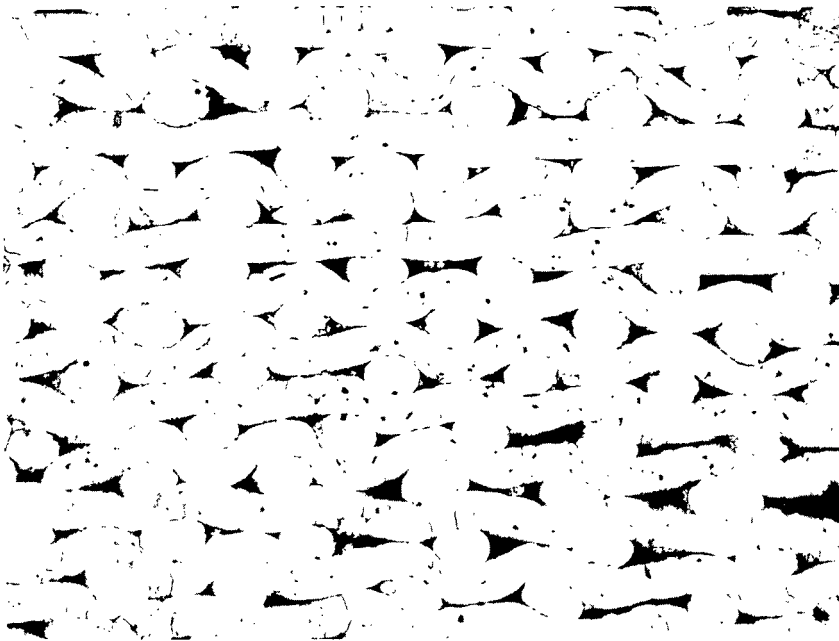
TABLE III. - Concluded. TENSILE DATA FOR INITIAL FOIL AND FIBERS, MATRIX, AND COMPOSITES

Composition	Condition	Test temperature		Ultimate tensile strength		Elongation, ^a percent	Reduction in area, percent
		°F	K				
				psi	N/m ²		
Properties of composites							
Tantalum, 5-volume- percent reinforcement	Rolled to 99-percent reduction in thickness ↓	77	298	200 000	13.79×10 ⁸	2	2
		1000	811	130 000	8.96	9	8
		1500	1089	61 000	4.21	4	24
		2200	1478	14 000	.96	27	54
Tantalum, 15-volume- percent reinforcement	Rolled to 95-percent reduction in thickness ↓	77	298	196 000	13.51×10 ⁸	3	2
		1000	811	155 000	10.69	14	5
		1500	1089	65 000	4.48	10	6
		2200	1478	15 000	1.03	24	58
	Rolled to 99-percent reduction in thickness	77	298	250 000	17.24×10 ⁸	2	2
Tantalum, 30-percent- percent reinforcement	Rolled to 80-percent reduction in thickness ↓	77	298	102 000	7.03×10 ⁸	11	36
		1000	811	60 000	4.14	18	28
		1500	1089	31 000	2.14	15	52
		2200	1478	16 000	1.10	17	50
	Rolled to 95-percent reduction in thickness	77	298	240 000	16.55×10 ⁸	1	1
	Rolled to 99-percent reduction in thickness Rolled to 99-percent reduction in thickness	77 77	298 298	322 000 297 000	22.20×10 ⁸ 20.48×10 ⁸	2 2	1 1

^aFrom crosshead motion.^bAt 2200° F (1478 K) for 3 hr.^cAt 1000° F (811 K) for 4 hr.^dAt 1500° F (1089 K) for 1/2 hr.

Microstructure

As-rolled condition. - Longitudinal and transverse microstructures of the composite sheet after a 28-percent overall reduction in thickness by rolling appear in figure 5. A transverse cross-sectional view of fibers interspersed with tantalum foil is shown in figure 5(a). Both the fibers and the foil appear to be approximately 0.0025 inch (6.35×10^{-5} m) thick in a direction perpendicular to the direction of rolling. A longitudinal section is shown in figure 5(b). The fibers and foil have recrystallized grains that are nonuniform in size. The grain boundaries are irregular in shape; they show no tendency to align themselves in the direction of rolling at this stage in reduction. A longi-



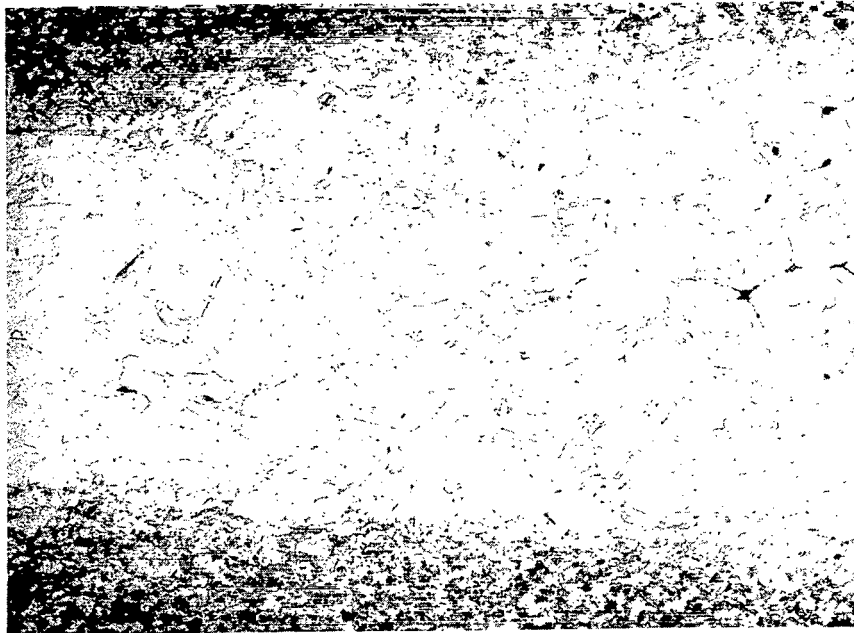
(a) Transverse.



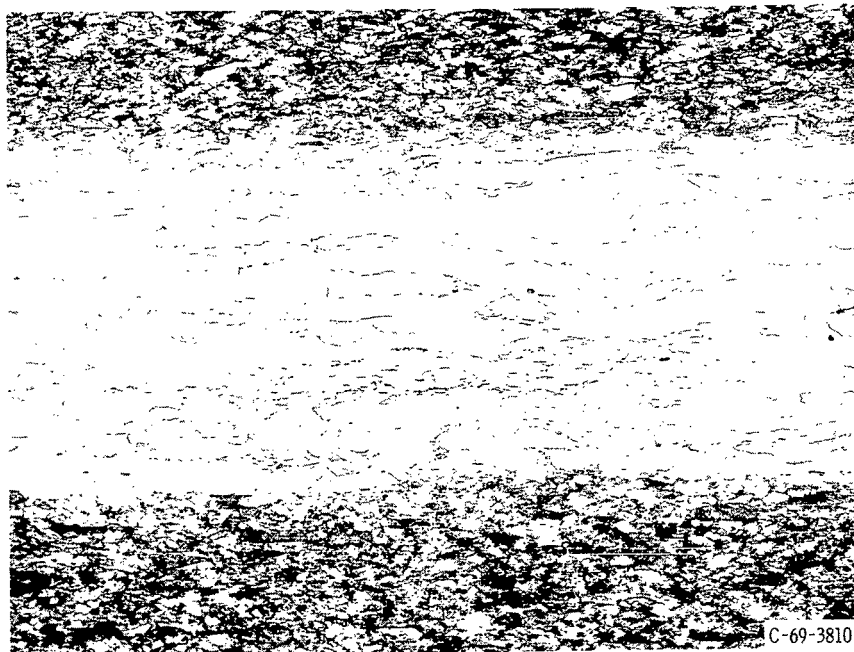
C-69-3809

(b) Longitudinal.

Figure 5. - Cross-sectional views of fibers interspersed with foil after 28-percent overall reduction in thickness by rolling. X75.



(a) Transverse.



C-69-3810

(b) Longitudinal.

Figure 6. - Cross-sectional views of composite after 54-percent overall reduction in thickness.
X75.

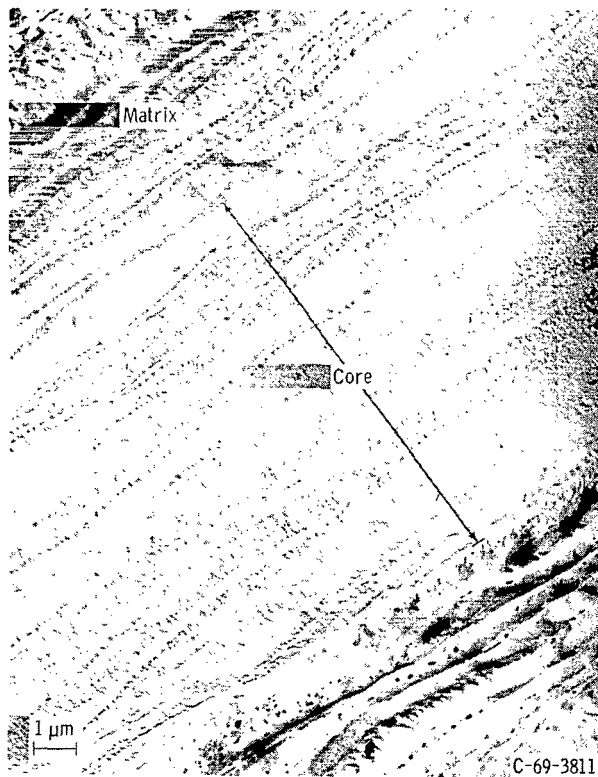


Figure 7. - Electron micrograph of 30-volume-percent composite specimen rolled to 99-percent reduction in thickness. X9500.

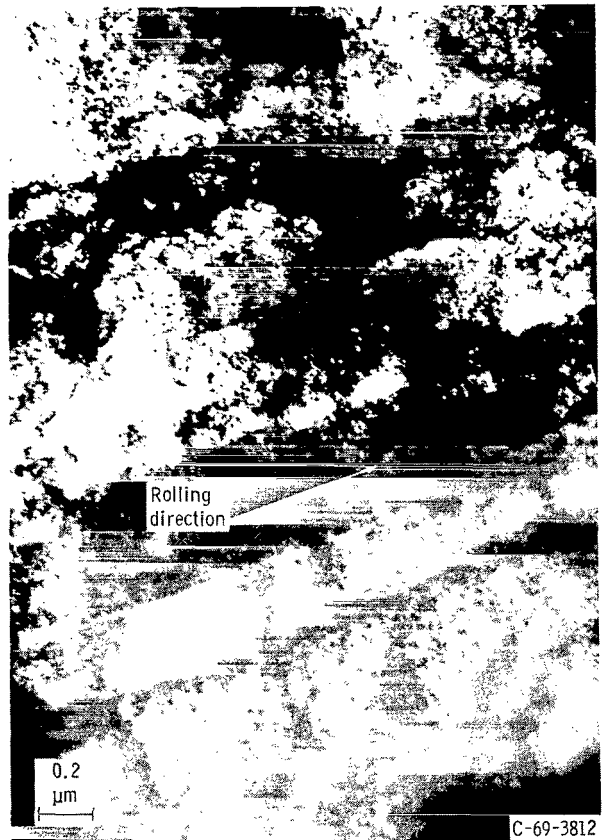


Figure 8. - Electron transmission view perpendicular to top surface of 30-volume-percent composite sheet specimen rolled to 99-percent reduction in thickness. X70 000.

tudinal and cross-sectional view of the composite after a 54-percent overall reduction in thickness are shown in figure 6. The transverse cross-sectional view (fig. 6(a)) reveals that the fibers were reduced to an oval shape which was approximately 0.0017 inch (4.32×10^{-5} m) thick. The void space appears to be almost completely eliminated. Figure 6(b) shows a longitudinal microstructure that indicates a slight tendency on the part of grain boundaries to align themselves parallel to the direction of rolling. Most of the grains still appear to be at least partially recrystallized. The microstructure displays enough distortion to suggest that some cold-work was retained.

Electron micrographs. - A typical 30-volume-percent composite specimen rolled to a 99-percent reduction in thickness is presented in figure 7. Shown is the central area of the sheet that consists entirely of fibers and foil before rolling. Rolling has produced a fibered structure with the fibers aligned parallel to the rolling direction. The width between subgrain boundary barriers is approximately 1 micrometer or less. The region outside the central zone is matrix material severely attacked by the etchant. Figure 8

is a photograph ($\times 70\,000$) made by electron transmission of the top view of the same composite specimen rolled in excess of a 99-percent reduction in thickness. The photograph shows a very high density of dislocations. A cellular arrangement exists, and there is occasional evidence of subgrain boundaries. The average cell dimension is well under a micrometer in the plane of the photograph normal to the rolling direction.

DISCUSSION

A high-strength composite material consisting of two forms of tantalum was produced. The material consolidated from tantalum wire, sheet, and powder was thermomechanically processed into sheet form. Actually, there are two general materials in the composites, both of which are tantalum based. One is the core made from the fibers of tantalum and tantalum foil, and the other is the matrix. The composite consisting of both these materials was thermomechanically worked.

In figure 9, the ultimate tensile strength of the composite is related to the volume percent of core at a test temperature of 77°F (298 K). The ultimate tensile strength was a linear function of volume percent core over the total range of reinforcement tested. The strength ranged from $173\,000\text{ psi}$ ($11.93 \times 10^8\text{ N/m}^2$) for the matrix to a maximum value of $322\,000\text{ psi}$ ($22.20 \times 10^8\text{ N/m}^2$) for the composite with a 30-volume-percent core. An extrapolation using the rule of mixtures indicated that the core material had a strength of approximately $650\,000\text{ psi}$ ($44.82 \times 10^8\text{ N/m}^2$). This strength is unusually high for tantalum.

Although the matrix powder with its residual tantalum oxide particles could be strengthened significantly by thermomechanical working, the core material, which ini-

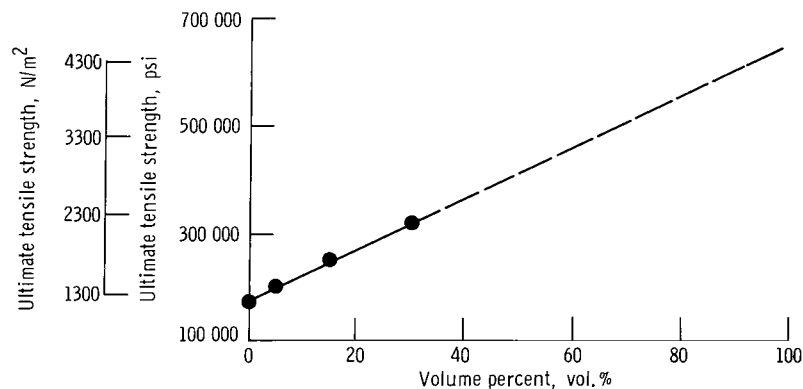


Figure 9. - Ultimate tensile strength as function of volume percent foil and fiber core at 77°F (298 K) for tantalum - tantalum-fiber composites after 99-percent reduction in thickness by rolling.

tially was relatively contaminant free, was strengthened more. There was more elongation of grain boundaries and creation of substructure in the core than in the powder product processed the same way (see fig. 7). The substructure in the core material was probably pinned by oxides generated during thermomechanical working. In the case of the dispersion-strengthened matrix material, however, the dispersoid coupled with the grain boundary would act as the inhibitor to subgrain growth. The considerable prior fabrication and work history apparently gave the fibers and foil of the core an advantage that resulted in the unusual strength of the core. It is suggested that the larger amounts of thermomechanical working greatly reduced major sources of failure such as grain boundaries and other crack instigators that were transverse to the applied stress. Figure 10 presents a comparison of the strengths of the matrix, the core (obtained by extrapolation, see fig. 9), a composite with a 30-volume-percent core, and unalloyed conventional tantalum sheet after rolling. The matrix, the composite, and the core strengths are higher than the value of 116 000 psi (8.0×10^8 N/m²) obtained for conventional tantalum sheet after rolling.

The high strength of the composite product may be attributed to a fine stable substructure where subgrain boundaries, or barriers, were aligned parallel to the major stress axis. It is known that tensile strength can be related to a barrier dimension. Such barrier dimensions may be controlled by prior work history. For example, it was shown with iron (ref. 2) that the spacing between barriers becomes important in deter-

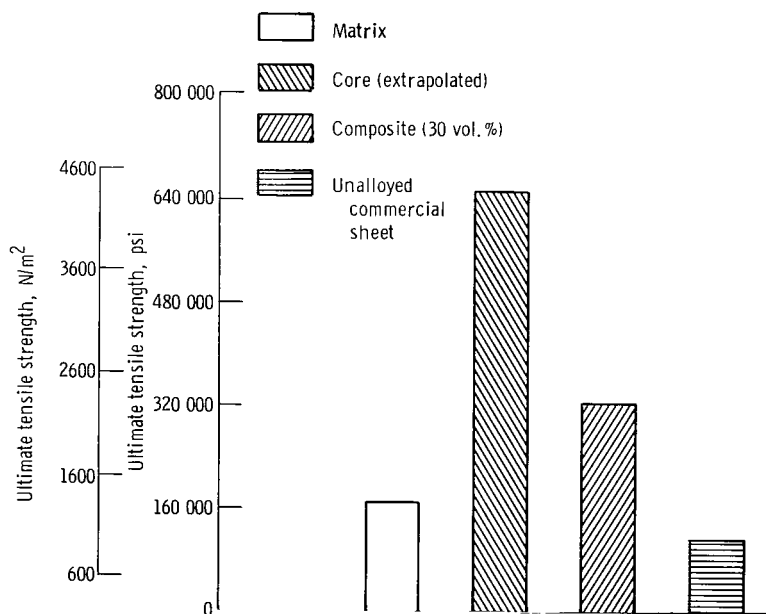


Figure 10. - Ultimate tensile strength at 77° F (298 K) after 99-percent reduction in thickness by rolling.

mining the proportional limit on flow stresses. Figures 7 and 8 make clear that the average cell, or barrier, dimension is well under a micrometer, which is small enough to cause considerable strengthening. This strength would not be expected to be retained to high temperatures because the cell-type substructure breaks down at temperatures of a few hundred degrees as a result of subgrain growth. However, there are many potential uses for high-strength tantalum at room temperature and at temperatures of a few hundred degrees. For example, the use of tantalum - tantalum-fiber sheet in place of pure tantalum could result in higher strength chemical containment vessels and stronger prosthetic devices in surgery.

CONCLUDING REMARKS

It is well known that tantalum has many aerospace uses, but most of these are high-temperature applications. Therefore, anything that is done to increase the high-temperature strength of tantalum is worthwhile. On the other hand, tantalum has some excellent uses for low-temperature applications, such as containment vessels for corrosive and explosive chemicals and prosthetic devices. Improving the strength of tantalum without lowering superior heat-transfer and resistance-to-corrosion capabilities can be very desirable, especially with regard to these low-temperature applications where high strength induced by working can be retained.

The high strength of the artificially made structure with fine barrier dimensions produced in this study may suggest a wide applicability of the substructure-strengthening effect, especially with respect to composites. With iron materials, a high-density dislocation network is created by thermomechanical working, and the network is stabilized by carbide diffusion and precipitation (ref. 2). The present investigation suggested that a high-density dislocation network produced by thermomechanical working and stabilized by oxygen diffusion and precipitation could account for the strength values obtained.

Putting a pure metal in contact with an impure metal while it is undergoing extensive mechanical work may be a general method to create a large degree of substructure that can be stabilized by an impurity as the substructure is formed. In this way, stable barrier dimensions can be produced that contribute to substantial strength gains.

The materials produced in this study point out another example in an already vast array of examples of how materials working in concert through composites can produce greatly improved properties for aerospace and conventional uses.

SUMMARY OF RESULTS

In an investigation of the effect of thermomechanical processing by rolling on the tensile strength and microstructural properties of a tantalum composite, the following results were obtained:

1. Tantalum sheet and fibers embedded in a matrix of tantalum powder were rolled to very high levels of reduction, which resulted in greatly improved strength at 77° F (298 K). A composite with 30-volume-percent sheet and fiber core reinforcement had a strength of 322 000 psi (22.2×10^8 N/m²). The strength of the composite core obtained by extrapolation was 650 000 psi (44.82×10^8 N/m²). The matrix strength was 173 000 psi (11.93×10^8 N/m²).

2. The high-strength level demonstrated by the composite at 77° F (298 K) was associated with a fine stable substructure generated by the thermomechanical working. Average cell, or barrier, dimensions were well under a micrometer, which is small enough to cause considerable strengthening.

3. The strength advantage of the composite over the matrix was retained to 1000° F (811 K). No difference in strength existed between the composite and matrix at 1500° and 2200° F (1089 and 1478 K).

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 17, 1969,
129-03.

REFERENCES

1. Sikora, Paul F.; and Hall, Robert W.: High-Temperature Tensile Properties of Wrought Sintered Tungsten. NASA TN D-79, 1959.
2. Embury, J. D.; and Fisher, R. M.: The Structure and Properties of Drawn Pearlite. Acta Met., vol. 14, no. 2, Feb. 1966, pp. 147-159.